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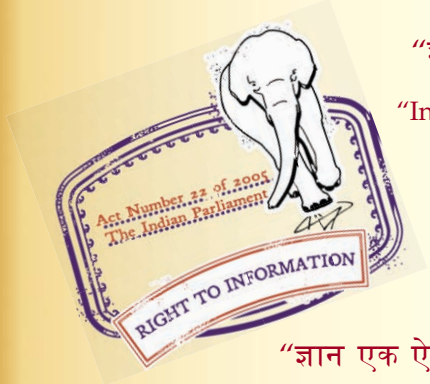
IS 1051 (1980): Pyrethrum Extracts [FAD 1: Pesticides and Pesticides Residue Analysis]



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IS : 1051 - 1980
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Indian Standard

SPECIFICATION FOR PYRETHRUM EXTRACTS

(Second Revision)

UDC 632·951 PYRETHRUM : 632·981·12

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1981

Indian Standard

SPECIFICATION FOR PYRETHRUM EXTRACTS

(Second Revision)

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Indian Standard

SPECIFICATION FOR PYRETHRUM EXTRACTS

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 28 October 1980, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 This standard was first published in 1957. The first version in 1973 incorporated two amendments of flash point and packing requirements. In this second version mercury reduction method has been incorporated for the determination of pyrethrins. A qualitative method based on thin layer chromatography (TLC) has also been incorporated.

0.3 Pyrethrum extracts containing varying percentages of pyrethrins are largely used in the control of insect pests of medical, agricultural and veterinary importance. They form as one of the insecticides in the household spray preparations because of quick knock down properties. They are prepared by extracting commercial pyrethrum flowers (*Chrysanthemum cinerariaefolium* Linn) with a mineral oil, such as kerosene and contain the ingredients occurring naturally in the flowers. The active principle in pyrethrum extract is composed of six esters, namely, pyrethrin I & II, cinerin I & II and jasmolin I & II. The method (*see* Appendix B) determines the strength of the extract by the determination of total of pyrethrin I + cinerin I + jasmolin I and denoting them as pyrethrin I and the total of pyrethrin II + cinerin II + jasmolin II and denoting them as pyrethrin II for the purpose of simplicity.

0.4 Pyrethrum extracts are generally manufactured to contain 2 percent (*m/m*) of total pyrethrins (*see* **0.3**).

0.5 In the preparation of this standard due consideration has been given to the provisions of the Insecticides Act, 1968 and the rules framed thereunder. However, this standard is subject to the restrictions imposed under these rules wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2 - 1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the method of sampling and test for pyrethrum extracts containing varying percentages of total pyrethrins.

2. REQUIREMENTS

2.1 Description and Identity

2.1.1 *Description* — The material shall be an extract of commercial pyrethrum flowers (*Chrysanthemum cinerariaefolium* Linn) in a mineral oil with or without minute quantity of added anti-oxidant but without a synergist. The material shall be homogeneous liquid. Sediment and/or suspended matter shall be negligible. It shall be free from synthetic pyrethroids.

2.1.2 *Identity* — The material shall comply with Identity Test as described under Appendix A and shall not contain any synthetic pyrethroids. If any spots, in addition to those given by the standard reference sample are detected, it should be ensured that these additional spots are not due to any synthetic pyrethroids. If the presence of any synthetic pyrethroid is established in the TLC testing, the total pyrethrin content (Appendix B) should not be determined as synthetic pyrethroid would analyse wrongly as pyrethrins.

2.2 Colour and Odour — The material shall be greenish yellow in colour and possess the characteristic odour of the commercial pyrethrum flowers.

2.3 Flash Point (Abel) — When determined by the method specified in IS : 1448 (P : 20) - 1960†, the flash point of the material shall not be below 32·0°C.

2.4 Total Pyrethrin Content — When determined by the method prescribed in Appendix B, the observed total pyrethrin content percent by mass, of any of the samples shall not differ from the nominal total

*Rules for rounding off numerical values (*revised*).

†Methods of test for petroleum and its products P : 20 Flash point by Abel apparatus.

pyrethrin content, percent by mass, by more than the tolerance limits indicated below:

Nominal Value, Percent	*Tolerance Limit, Percent*	
Up to 9	+ 10 — 5	of the nominal value
Above 9 and below 50	± 5	
50 and above	+ 5 — 3	

2.4.1 The actual value of total pyrethrin content in the material shall be calculated to the second decimal place and then rounded off to the first decimal place before applying tolerances given in **2.4.**

2.4.2 The average content of all samples taken shall not be lower than the nominal content.

2.5 Additional Requirement for the Material for Use as an Aerosol — The matter insoluble in dichlorodifluoromethane in the material meant for use as an aerosol, shall not exceed 1·5 percent by mass when determined by the method prescribed in Appendix C.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as per requirements given in IS : 8190 (Part II) - 1980*.

3.2 Marking — The containers shall bear legibly and indelibly the following information and any other information as is necessary under the Insecticides Act and Rules:

a) Common name of the material,

b) Name of the manufacturer,

c) Date of manufacture,

d) Batch number,

e) Net volume of contents,

f) Nominal total pyrethrin content (*m/m*), and

g) The minimum cautionary notice as worded in the Insecticides Act and Rules.

3.2.1 The containers may also be marked with the ISI Certification Mark.

*Requirements for packing of pesticides : Part II Liquid pesticides (*first revision*).

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in the 'Indian Standard methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the samples shall be drawn as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out by the appropriate methods referred to in **2.1.2** and **2.3** to **2.5**.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070 - 1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Clause* 2.1.2)

IDENTITY TEST FOR PYRETHRINS

A-0. PRINCIPLE

A-0.1 The method is based on the thin layer chromatographic separation of pyrethrins from the added synthetic pyrethroids, if any.

A-1. APPARATUS

A-1.1 TLC Plates — standard TLC glass plates coated with 0·38 mm thick layer of Silica Gel HF 254.

*Specification for water for general laboratory use (*second revision*).

A-1.2 Accessories

A-1.2.1 *Applicator* — standard.

A-1.2.2 *Pipettes* — 5 microlitres.

A-1.2.3 *Hair Drier or Any Other Arrangement to Provide Hot Air*

A-1.2.4 *Developing Tank* — of a suitable size and covered on the inside with filter paper for saturation. The tank to be provided with a lid to prevent loss of solvent by evaporation.

A-1.2.5 *Iodine Staining Jar* — glass.

A-1.2.6 *TLC Spotting Guide* — standard.

A-2. REAGENTS

A-2.1 Developing Solvent — Benzene : Ethyl acetate (95 : 5 *v/v*).

A-2.2 Iodine-Crystals

A-2.3 Methyl Alcohol

A-2.4 Standard Pyrethrum Oleoresin — of known total pyrethrin content.

A-2.5 Petroleum Ether (40 - 60°) bp

A-2.6 Synthetic Pyrethroid

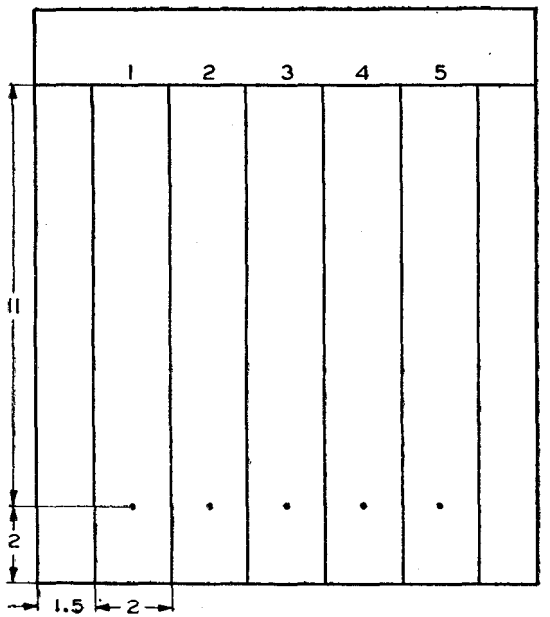
A-3. PROCEDURE

A-3.1 Preparation of Standard for TLC — Dilute the standard pyrethrum oleoresin of known pyrethrin content, with mineral turpentine oil to the same nominal total pyrethrin content as expected in the sample. Take 10 ml of this diluted standard in a 250 ml separating funnel. Add 20 ml petroleum-ether and 10 ml methyl alcohol. Shake for 2 minutes and allow the layers to separate. Remove the lower methanol layer into a 100-ml dry stoppered cylinder. Repeat the extraction of the material in the separating funnel twice more using 10 ml methanol each time and removing the methanol layer in the same 100-ml stoppered cylinder. Make the volume in the stoppered cylinder to 100 ml with methanol. Call this STANDARD FOR TLC. Best results are obtained when the STANDARD FOR TLC is a 0·2% (*m/v*) solution of total pyrethrins in methanol.

A-3.2 Preparation of Sample for TLC — Extract 10 ml of the sample with methanol exactly in the same manner as given under **A-3.1**. Call this SAMPLE FOR TLC.

A-3.3 Thin Layer Chromatography — Place the developing solvent in the developing tank so that layer of the solvent is 1·5 cm from the bottom of the tank. Cover with lid and allow to stand for 1 hour.

A-3.3.1 Using a template, mark the TLC plate with a sharp instrument as shown in Fig. 1. Keep the marked plate in an oven at 110°C for 15 minutes and then allow it to cool to room temperature.



All dimensions in centimetres.

FIG. 1 TLC FOR PYRETHRINS

A-3.3.2 Spot 5 microlitres of each of STANDARD FOR TLC and SAMPLE FOR TLC on glass strips 1 and 2 taking care to see that the spots are at a distance of 2 cm from the bottom. To prevent the spots from spreading, use hot air to blow off the solvent after each spotting. Similarly, in strip 3 next, first spot 5 microlitres of STANDARD FOR TLC and then superimpose 5 microlitres of SAMPLE FOR TLC on the same spot.

A-3.3.3 Allow the solvent from the spots to evaporate and then keep this plate in the developing tank. Allow the solvent front to reach the top line and then remove the plate and allow the solvent to evaporate.

Keep the plate in iodine vapours. Spots are properly developed after one hour in iodine vapours.

A-4. OBSERVATIONS AND INFERENCES

A-4.1 The sample shall be considered free from synthetic pyrethroids like allethrin if the chromatograms of the standard, the sample and the superimposed mixture are exactly alike.

A-4.2 If any additional spot/spots are seen in the chromatogram of the sample, the TLC shall be repeated using solution of synthetic pyrethroids like allethrin in methanol (0·2% *m/v*) for comparison. The standard, the sample, the synthetic pyrethroid and the mixture of standard + synthetic pyrethroid and the mixture of sample + synthetic pyrethroid should be used.

A-4.3 If the sample contains synthetic pyrethroid like allethrin, the additional spot/spots would correspond to the spot/spots of the synthetic pyrethroid indicating the presence of synthetic pyrethroid like allethrin in the sample. In such a case, the product shall be rejected.

APPENDIX B

(*Clauses* 2.1.2 *and* 2.4)

DETERMINATION OF TOTAL PYRETHRIN CONTENT

B-0. PRINCIPLE

B-0.1 The pyrethrins after extraction are hydrolysed to chrysanthemum mono- and dicarboxylic acids. Monocarboxylic acid from 'Pyrethrin I', after extraction is reacted with Deniges reagent and mercurous sulphate formed, is titrated with potassium iodate. Dicarboxylic acid from Pyrethrin II, remaining after the extraction of monocarboxylic acid is extracted and titrated with standard sodium hydroxide.

B-1. REAGENTS

B-1.1 Deniges Reagents — Mix 5 g yellow mercuric oxide with 40 ml water, and while stirring, slowly add 20 ml sulphuric acid, then add additional 40 ml water and stir until all dissolves. Test for the absence of mercurous mercury by adding a few drops of iodine monochloride solution to 10 ml and titrating with potassium iodate standard solution as in **B-2.2**.

B-1.2 Iodine Monochloride Solution — Dissolve 10 g potassium iodide and 6·44 g potassium iodate in 75 ml water in glass stoppered bottle,

add 75 ml hydrochloric acid and 5 ml chloroform and adjust to faint iodine colour (in chloroform) by adding dilute potassium iodide or potassium iodate solution. If much iodine is liberated, use stronger solution of potassium iodate than 0·01 M at first, making final adjustment with 0·01 M solution. Keep in dark and readjust when necessary. It should not be stored in refrigerator.

B-1.3 Potassium Iodate Standard Solution — 0·01 M. Dissolve 2·14 g pure potassium iodate, previously dried at 105°C, in water and dilute to one litre. One ml of solution = 0·005 7 g pyrethrin I.

B-1.4 Alcoholic Sodium Hydroxide Solution — 1 N and 0·5 N.

B-1.5 Petroleum Ether — aromatic free, boiling range 40-60°C or 60-80°C.

B-1.6 Ethyl Ether — peroxide free.

B-1.7 Sodium Hydroxide Standard Solution — 0·02 N.

B-1.8 Filter Cel

B-1.9 Barium Chloride Solution — 10 percent (*m*/*v*).

B-1.10 Dilute Sulphuric Acid — 1 : 4 (*v*/*v*).

B-1.11 Ethyl Alcohol — 95 percent (*v*/*v*) and 99 percent (*v*/*v*), anhydrous.

B-1.12 Sodium Chloride — solid as well as saturated solution.

B-1.13 Chloroform

B-1.14 Dilute Hydrochloric Acid—3 : 2 (*v*/*v*).

B-1.15 Phenolphthalein Indicator Solution — one percent (*m*/*v*) in ethyl alcohol.

B-2. PROCEDURE

B-2.0 Weigh sample containing 40-150 mg total pyrethrins, add 50 ml petroleum ether and 1 g filter cel, and place in refrigerator at 0 ± 0·5°C overnight. Filter through Gooch into 300 ml Erlenmeyer flask and wash with three 15 ml portions of cold petroleum ether. Evaporate filtrate and washings on water bath, using air current until the petroleum ether is almost entirely removed.

B-2.1 Add 20 ml 1 N alcoholic sodium hydroxide, or more if necessary, to extract pyrethrins, connect to reflux condenser, and boil gently for 60 to 90 minutes. Transfer to 600-ml beaker and add enough water to make aqueous layer 200 ml. If more than 20 ml alcoholic sodium

hydroxide solution was used, add enough water so that all alcohol is removed when volume is reduced to 150 ml. Add few glass beads and boil aqueous layer down to 150 ml. Transfer to 500-ml separator and drain aqueous layer into 250-ml volumetric flask. Wash oil layer once with water and add washings to aqueous portion. If slight emulsion still persists after draining aqueous layer and washings, add two to three ml 10 percent barium chloride solution but do not shake vigorously after adding barium chloride, because reversed emulsion difficult to separate may form. To aqueous solution in 250-ml flask add 1 g filter cel and approximately 10 ml of the barium chloride solution. Swirl gently and let stand for 30 minutes. Dilute to volume; mix thoroughly and filter off 200 ml. Test filtrate with barium chloride solution to see if enough has been added to obtain clear solution. Neutralize with sulphuric acid (1 : 4), using 1 drop phenolphthalein and add 1 ml excess. (If necessary to hold solution overnight at this point, leave in alkaline condition.)

B-2.2 Determination of Pyrethrin I — Filter acid solution from **B-2.1** through 7 cm paper, coated lightly with suspension of filter cel in water, on buchner, and wash with three 15 ml portions of water. Transfer to 500-ml glass stoppered separator and extract with two 50 ml portions petroleum ether. Shake each extract approximately for one minute, releasing pressure if necessary by inverting separator and carefully venting through stopcock. Let layers separate for approximately five minutes or until aqueous layer is clear before draining and re-extraction. Reserve aqueous layer for pyrethrin II determination. Do not combine petroleum ether extracts but wash each in sequence with same three 10 ml portions water, and filter petroleum ether extracts through small cotton plug, into a clean 250-ml separator. Wash separators and cotton in sequence with 5 ml petroleum ether. Extract combined petroleum ether solutions with 5 ml 0·1 N sodium hydroxide, shaking vigorously for approximately one minute. Let layers separate for approximately 5 minutes before draining aqueous layer into 100-ml beaker. Wash petroleum ether with additional 5-ml portion 0·1 N sodium hydroxide and with 5 ml water, adding washings to beaker. Add 10 ml Deniges reagent and let stand in complete darkness for one hour at 25 $\pm$ 2°C. Add 20 ml alcohol and precipitate mercurous chloride with 3 ml saturated sodium chloride solution. Warm to approximately 60°C and let stand several minutes until precipitate coagulates and settles. Filter through small paper, transferring all precipitate to paper, and wash with approximately 10 ml hot alcohol. Wash with 2 or more 10 ml portions hot chloroform and place paper and contents in 250-ml glass stoppered conical flask. Add 50 ml cooled dilute hydrochloric acid (3 : 2). Add 5 ml chloroform and 1 ml freshly adjusted iodine monochloride solution and titrate with standard potassium iodate solution, shaking vigorously approximately 30 second after each addition, until no iodine colour

remains in chloroform layer. Take as end point when red colour disappears from solvent layer and does not return within three minutes. From standard potassium iodate solution used in titration and blank on Deniges reagent, calculate percent pyrethrin I.

B-2.3 Calculation — 1 ml of 0·01 M KIO_3 = 0·005 7 g pyrethrin I.

NOTE — Chrysanthemum monocarboxylic acid reacts with Deniges reagent to form series of colours beginning with phenolphthalein red, which gradually changes to purple, then to blue and finally to bluish green. Colour reaction is very distinct with 5 mg monocarboxylic acid and amounts as low as 1 mg can usually be detected. Therefore, no pyrethrin I should be reported if colour reaction is negative. With samples containing much perfume or other saponifiable ingredients, it may be necessary to use as much as 50 ml 1 N alcoholic sodium hydroxide. When lethanes are present, after washing mercurous chloride precipitate with alcohol and chloroform, wash once more with alcohol and then several times with hot water.

B-2.4 Determination of Pyrethrin II — If necessary, filter aqueous residue reserved in **B-2.2** from petroleum ether extract through Gooch. Concentrate filtrate to approximately 50 ml and transfer to 500-ml glass stoppered separator. Wash beaker with three 15 ml portions water. Acidify with 10 ml hydrochloric acid and saturate with sodium chloride. (Acidified aqueous layer must contain visible sodium chloride crystals throughout following extractions.)

Extract with 50-ml ether, drain aqueous layer into second separator and extract again with 50 ml ether. Continue extracting and draining aqueous layer, using 35 ml for third and fourth extractions. Shake each extract for approximately one minute, releasing pressure, if necessary, by inverting separator and carefully venting through stopcock. Let layers separate for approximately five minutes or until aqueous layer is clear before subsequent draining and extraction. Combine ether extracts, drain and wash with three 10 ml portions saturated sodium chloride solution. Filter ether extracts through cotton plug into 500-ml conical flask and wash separator and cotton with additional 10 ml ether. Evaporate ether on water bath, and remove any fumes of hydrochloric acid with air current and continue heating for approximately five minutes. Dry for 10 minutes at 100°C. Add 2 ml neutral alcohol and 20 ml water and heat to dissolve acid. Cool, filter through Gooch if necessary, add two drops phenolphthalein and titrate with 0·02 N sodium hydroxide. Check normality of 0·02 N sodium hydroxide on the same day, as the sample is titrated.

B-2.5 Calculation

1 ml of 0·02 N NaOH = 0·003 74 g pyrethrin II.

APPENDIX C

(*Clause* 2.5)

DETERMINATION OF MATTER INSOLUBLE IN DICHLORODIFLUOROMETHANE

C-1. APPARATUS

C-1.1 The apparatus is shown in Fig. 2. It consists of a strong glass bottle *A* fitted with a pressure cap *B* and needle valve *C*. The bottle is held in position by means of a frame *F* constructed from two steel rods. By means of lock nuts the lower ends of the steel rods are screwed to the frame base *J* which also holds the bottom of the bottle. A rubber cushion *K* is placed between the bottom of the bottle and the frame

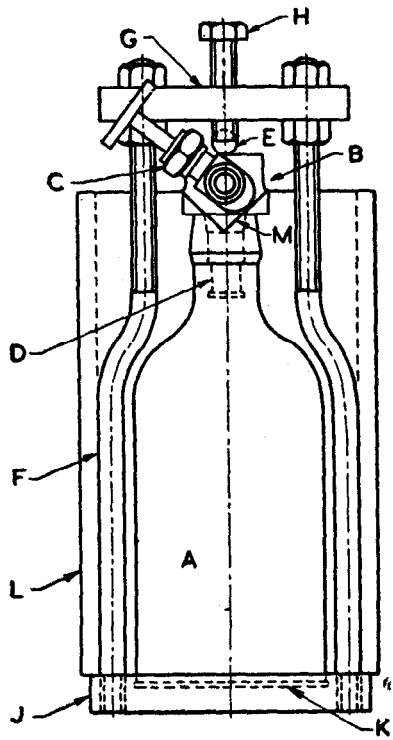


FIG. 2 APPARATUS FOR THE DETERMINATION OF MATTER INSOLUBLE IN DICHLORODIFLUOROMETHANE

base. The upper ends of the steel rods are screwed to an adjustable cross bar *G* which also carries a tightening screw *H* in the centre. The pressure cap is held tightly on the bottle by the adjustable cross bar by means of the tightening screw, a ball-bearing *E* being used between the screw and the pressure cap. The ball bearing is used to obtain a uniform pressure on the neoprene washer *N* (*see* Fig. 3) inside the top of the bottle. Correct adjustment of the height of the cross bar is made by placing the bottle in the frame turning the lock nuts to obtain sufficient clearance for removing the bottle. The bottle, together with the frame, is housed in a safety shield *L*. The various parts of the apparatus are as follows.

C-1.1.1 *Glass Bottle, A* — Sufficiently strong and of the shape shown in Fig. 2.

C-1.1.2 *Pressure Cap, B* — Constructed from a 30 mm round brass stock by machining on a turning lathe to the same shape as the glass stopper furnished with the bottle. The lower end of the cap is then

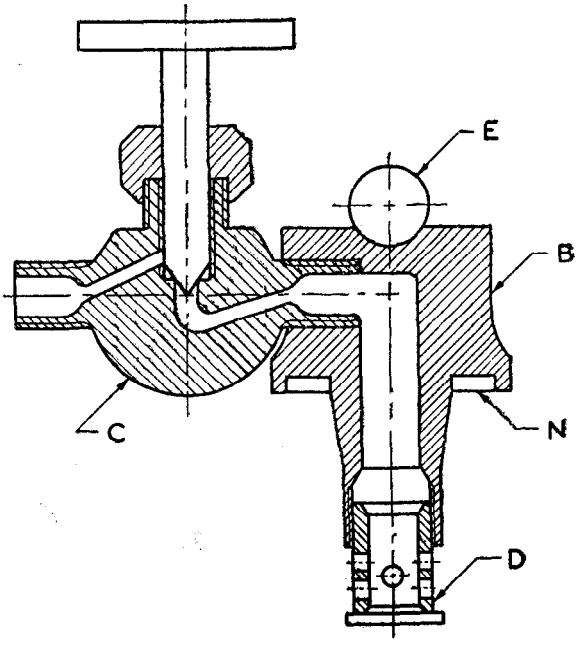


FIG. 3 VERTICAL SECTION OF PRESSURE CAP FITTED WITH NEEDLE VALVE AND FILTER SCREEN

drilled to a depth of 30 mm with a 5 mm drill, and then at right angles on the side for the outlet to the needle valve *C*. The lower end of the cap is then drilled to a depth of 5 mm with an 8-mm drill. The opening is threaded on the lathe to fit the filter screen *D* commonly used on oil-burner tips. The side outlet is drilled to a depth of 9·5 mm with an 8-mm drill and threaded with a 3-mm pipe tap to fit the needle valve *C*. A depression is made in the centre of the top of the cap to hold a 9·5-mm ball-bearing *E*. The vertical section of the pressure cap *B* fitted with the needle valve *C* and the filter screen *D* is shown in Fig. 3.

C-1.1.3 *Frame, F* — constructed from two 9·5-mm steel rods threaded on both ends. The upper end of each rod is threaded for a distance of 5 cm to permit adjustment of the cross bar *G* for bottles of different heights. The lower end of each rod is also threaded to a convenient length for screwing into the holes made in the frame base *J*. The rods are heated to a cherry red and bent in the shape shown in Fig. 2.

C-1.1.4 *Cross Bar, G* — made from 13 × 32 mm brass stock. Two holes of 9·5 mm diameter are drilled in the cross bar 5 cm apart. Another hole to permit the tightening screw is drilled at equal distance from the other two holes with an 8-mm drill and threaded with a 9·5-mm tap.

C-1.1.5 *Tightening Screw, H* — of the size meant for the central hole in the cross bar *G*. The end of the screw is recessed with 9·5-mm drill to hold the ball-bearing *E*.

C-1.1.6 *Frame Base, J* — of diameter 102 mm made from brass stock. The thickness of the frame base is 13 mm. A depression, 5 mm deep and 70 mm in diameter, is made in the centre of the frame base to hold the bottom of the bottle *A* in place. Two 8-mm holes are drilled diametrically opposite outside the depression in the frame base and threaded with 9·5-mm tap to receive the lower ends of the two steel rods.

C-1.1.7 *Rubber Cushion, K* — a 70-mm disc cut from a 3-mm thick rubber sheet.

C-1.1.8 *Safety Shield, L* — made by cutting a 178-mm section from a 95-mm Lucite tubing. A 38-mm notch *M* is cut in the tubing, to make it possible to adjust the needle valve *C* and still have the shield extended above the level of the bottle *A* for maximum protection.

C-2. REAGENTS

C-2.1 Acetone

C-2.2 Ethyl Alcohol Sulphuric Acid Mixture — 1 : 9 (*v*/*v*). Mix together one volume of ethyl alcohol and 9 volumes of concentrated sulphuric acid.

C-2.3 Dichlorodifluoromethane

C-2.4 Chloroform

C-3. PROCEDURE

C-3.1 Preparing the Apparatus — Fill the filter screen *D* with lamb's wool. Wash the filter screen, the pressure cap *B* and the needle valve *C* first with acetone and then with chloroform. Clean the bottle *A* with ethyl alcohol-sulphuric acid mixture and then rinse several times with water. Dry the bottle, filter screen, pressure cap and the needle valve in an oven at 105 ± 1°C for one hour and cool in a desiccator.

C-3.2 Weigh accurately about 6 g of the material into the glass bottle *A* and place it in its position in the apparatus. Fit the filter screen *D*, the needle valve *C* and the neoprene washer *N* to the pressure cap *B* and place in the top of the bottle. Place the ball-bearing *E* between the tightening screw *H* and the pressure cap *B* and tighten the screw. Place the safety shield *L* over this assembled unit. Add 294 ± 1 g of liquid dichlorodifluoromethane to the bottle *A* by following the method given under **C-3.2.1**.

C-3.2.1 Open the needle valve *C*, connect it to a vacuum pump and evacuate the bottle *A* to at least 63·5 cm of mercury. Close the needle valve *C* and disconnect the vacuum pump. Place the assembly of the apparatus after evacuation on a suitable balance. Connect the needle valve *C* to the source of liquid dichlorodifluoromethane by means of a suitable hose, counterpoise the apparatus by placing necessary weight on the other pan of the balance and then place additional weight equivalent to 294 g on it. Open the needle valve *C* to allow dichlorodifluoromethane flow into the bottle *A* and close it when the necessary quantity has been added. Disconnect the source of dichlorodifluoromethane and remove the hose from the needle valve *C*.

C-3.3 Place the assembly of the apparatus, after adding dichlorodifluoromethane, in a rack so as to allow the bottle *A* to rest at an angle of 30°. At intervals of 10 minutes over a period of two hours, rotate the assembly of the apparatus in the rack to approximately 45°. Allow the assembly of the apparatus to remain in the rack overnight. Remove the dichlorodifluoromethane by holding the needle valve *C* downward and releasing the liquid slowly by opening the needle valve. When all the liquid has been discharged, weigh 150 ± 1 g of dichlorodifluoromethane into the bottle *A* by the method used earlier, rinse the contents of the bottle by shaking and completely remove the dichlorodifluoromethane as before.

C-3.4 Remove the pressure cap *B* from the bottle *A* and detach the filter screen *D* from it. Place the filter screen in a 10-ml beaker, add 5 to 7 ml of chloroform and allow to stand. Add 15 ml of chloroform to the bottle *A* and rotate while holding it in a horizontal position. Transfer the contents of the bottle to a tared 125-ml Erlenmeyer flask. Wash the bottle four times with 15 ml portions of chloroform and transfer each washing to the Erlenmeyer flask. Transfer the chloroform contained in the 10-ml beaker to the Erlenmeyer flask. Wash the beaker and the filter screen *D* with a few 5-ml portions of chloroform and transfer the washings to the Erlenmeyer flask either by distillation or by heating slowly on a steam-bath. Place the flask in an oven maintained at 105 ± 2°C and dry the residue for one hour. Cool the flask containing the dry residue in a desiccator for two hours and weigh. Note the mass of the dry residue contained in the Erlenmeyer flask.

C-4. CALCULATION

Matter insoluble in dichlorodifluoromethane, in the material, percent by mass $= \frac{100\,m}{M}$

where

m = mass, in g, of the dry residue (*see* **C-3.4**); and

M = mass, in g, of the material taken for the test (*see* **C-3.2**).

(*Continued from page* 2)

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AMENDMENT NO. 1 APRIL 1985

TO

IS:1051-1980 SPECIFICATION FOR PYRETHRUM EXTRACTS

(Second Revision)

(*Page 6, clause 4.1 and Note*) - Substitute the following for the existing matter:

'4.1 Representative samples of the material shall be drawn as prescribed in IS:10627-1983 Methods for sampling of pesticidal formulations.'

(*Page 7, clause A-2.6*) - Substitute the following for the existing matter:

'A-2.6 Synthetic Pyrethroid - Allethrin and others.'

(*Page 8, clause A-3.3.2*) - Add the following new matter at the end:

'In strip 4 next, similarly spot allethrin or other synthetic pyrethroids in methanol (0.2% *m/v*). In the last strip 5, spot 5 µl of standard for TLC and then superimpose 5 µl of allethrin or other synthetic pyrethroids on the same spot.'

(*Page 8, clause A-3.3.3*) - Substitute the following for the existing matter:

'A-3.3.3 Allow the solvents from the spots to evaporate and keep this plate in the developing tank at 28° to 35°C. Allow the plate to develop for about 40 minutes when the solvent front reached the top line uniformly. Remove the plate and allow the solvents to evaporate completely by keeping it at about 80°C for 10 minutes. Keep the plate in iodine vapours when the spots are developed after an hour.'

(*Page 9, clause A-4.1*) - Substitute the following for the existing matter:

'The sample shall be considered genuine and free from synthetic pyrethroid allethrin or others, if chromatogram in strip 1, 2 and 3 are identical and also differ from the chromatogram in strip 5 as regards additional spot/spots (including elongated merged spots). Presence of allethrin or other synthetic pyrethroid is confirmed by additional spot/spots (including elongated merged spot/spots) in case of chromatogram of strip 2, 3 and 5 which also corresponds to the spot/spots of synthetic pyrethroid in strip 4. The sample will be rejected if allethrin or other synthetic pyrethroid is present.'

(*Page 9, clauses A-4.2 and A-4.3*) - Delete.

AMENDMENT NO. 2 NOVEMBER 1987

TO

IS:1051-1980 SPECIFICATION FOR PYRETHRUM EXTRACTS

(Second Revision)

[Page 8, clause A-3.3.2 (see also Amendment No. 1] - Substitute the following for the existing clause:

'A-3.3.2 Spot 5 µl of each of STANDARDS FOR TLC and SAMPLE FOR TLC on glass strips 1 and 2 taking care to see that the spots are at a distance of 2 cm from the bottom. To prevent the spots from spreading, use hot air to blow off the solvent after each spotting. Similarly, in strip 3 next, first spot 5 µl of STANDARD FOR TLC and then superimpose 5 µl of SAMPLE FOR TLC on the same spot. In strip 4 next, similarly spot allethrin or other synthetic pyrethroids in methanol (0.2% m/v). In the strip 5, spot 5 µl of standard for TLC and then superimpose 5 µl of allethrin or other synthetic pyrethroids on the same spot. In last strip 6 spot 5 µl of sample and then superimpose 5 µl of allethrin or other synthetic pyrethroids.'

[Page 9, clause A-4.1 (see also Amendment No. 1] Substitute the following for the existing clause:

'A-4.1 The sample shall be considered genuine and free from synthetic pyrethroid allethrin or others, if chromatogram in strip 1, 2 and 3 are identical and also differ from the chromatogram in strip 5 and

6 as regards additional spot/spots (including elongated merged spots). Presence of allethrin or other synthetic pyrethroid is conformed by additional spot/spots (including elongated merged spot/spots) in case of chromatogram of strip 2 and 6 are identical. The sample will be rejected if allethrin or other synthetic pyrethroid is present.'

(AFCDC 6)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 3 MAY 1994
TO
IS 1051 : 1980 SPECIFICATION FOR PYRETHRUM EXTRACTS

(Second Revision)

(*Page* 6, *clause* **4.1**) — Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture, sampling shall be done as prescribed in IS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause **2.4** of the standard.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 4 DECEMBER 2006
TO
IS 1051 : 1980 SPECIFICATION FOR PYRETHRUM EXTRACTS

(Second Revision)

(*Page* 10, *Appendix* B, *clause* **B-1.8**) — Substitute '**Filter Cell** — Filtering aid available commercially in the form of powder to facilitate filtration1' *for* '**Filter Cel**'.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 5 SEPTEMBER 2008
TO
IS 1051 : 1980 SPECIFICATION FOR PYRETHRUM EXTRACTS

(Second Revision)

(*Page* 5, *clause* **2.4.2**) — Insert the following new clause after **2.4.2** and renumber the existing clause **2.5** as **2.6**:

'**2.5 Saponification Value** — When determined by the method specified in **15** of IS 548 (Part 1) : 1964, the Saponification value of Pyrethrum extract shall not exceed 10.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 6 DECEMBER 2010
TO
IS 1051 : 1980 SPECIFICATION FOR PYRETHRUM EXTRACTS

(Second Revision)

(*Page* 4, *clause* **2.2**) — Substitute the following for the existing:

'**2.2 Colour and Odour** — The material shall possess the characteristic odour of the commercial pyrethrum flowers. When determined by the method prescribed in Appendix D, the optical density of the material should be less than 0.900.'

(*Page* 17, *Appendix* C, *clause* **C-4**) — Add the following new Appendix after **C-4**:

'APPENDIX D
(*Clause* 2.2)

DETERMINATION OF COLOUR

D-0 PRINCIPLE

The natural colouring matter content of pyrethrum extract is extracted with mineral turpentine oil. The absorbance of the solution obtained is measured by using a UV-Visible Spectrometer at a wavelength of 435 nm.

D-1 APPARATUS

D-1.1 UV-Visible Spectrophotometer suitable for measuring at wavelength of 435 nm and having a band width of 2 nm.

D-1.2 Volumetric Flask, 20 ml capacity.

D-1.3 Measuring Cylinder

D-1.4 Cuvettes

D-2 REAGENTS

D-2.1 Solvent, mineral turpentine oil of known purity.

D-3 PROCEDURE

Take 1 ml of the pyrethrum extract in the 20-ml volumetric flask. Add to it mineral turpentine oil and make up the volume up to the mark with mineral turpentine oil. Shake the flask to mix the contents thoroughly. Take a pair of clean cuvettes. Switch on the Spectrophotometer and set the wavelength at 435 nm. In one cuvette take the solvent and place this cuvette in the reference cell of the spectrophotometer. Take the diluted sample from the volumetric flask in the other cuvette and place the cuvette in the sample cell of the spectrophotometer. Close the chamber and measure the absorbance at 435 nm.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India